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THE MEASUREMENT OF RESIDUAL STRESS IN URANIUM
USING ENERGY DISPERSIVE X-RAY DIFFRACTION

Final Technical Report

by

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Energy dispersive X-ray diffraction using synchrotron radiation was used to perform residual stress measurements on a bar of uranium. The basic theory of energy dispersive diffraction is presented, and the features most relevant to the work reported here are discussed. Residual stress measurements were made using the sin²ψ method which is described in detail.

Despite using the higher energy radiation produced by the 5 Tesla wiggler of the UK Synchrotron Radiation Source (SRS), the penetration was insufficient to measure inside the bulk of material. Our results therefore apply to a thin surface layer which is a mixture of uranium and uranium oxide. Within this surface, we found a residual compressive stress of 1097 MPa. The accuracy of our measurements was limited by the inability to use transmission geometry as a result of the high absorption. In energy dispersive diffraction, reflection geometry with high energy radiation requires low angles of diffraction which limit the range over which the sample can be tilted to perform stress measurements. These points are discussed in some detail in our conclusion where suggestions for future work are made.

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SUMMARY

We have used energy dispersive diffraction and synchrotron radiation to perform residual stress measurements on a bar of uranium metal. The basic theory of energy dispersive diffraction is presented and the features most relevant to the work reported here are discussed. The residual stress measurements were made using the 'sin$^2 \psi$' method which is described in some detail.

We present the results obtained in this preliminary study of the feasibility of the technique. Despite using the higher energy radiation produced by the 5 Tesla wiggler of the UK Synchrotron Radiation Source (SRS), the penetration was insufficient to measure inside the bulk of the material. Our final results therefore only apply to a thin surface layer which is a mixture of uranium and uranium oxide. Within this surface, we found a residual compressive stress of 1097 MPa. The accuracy of our measurements was limited by the inability to use transmission geometry as a result of the high absorption. In energy dispersive diffraction, reflection geometry with high energy radiation requires low angles of diffraction which limit the range over which the sample can be tilted to perform stress measurements. These points are discussed in some detail in our conclusion where suggestions for future work are made.

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Energy dispersive diffraction. synchrotron radiation. wiggler. residual stress. 'sin$^2 \psi$' method. uranium

NOTE

The work reported here was carried out using energy dispersive diffraction and high energy synchrotron radiation produced by a 5 Tesla wiggler. Readers requiring more information on these topics should consult the final technical report on *High Pressure Studies Using Energy Dispersive Diffraction of High Energy X-Rays* (U.S. Army Contract Number : DAJA 45-83-C-0031).

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1 - Introduction

In the work reported here we have used fixed angle Energy Dispersive X-ray Diffraction (EDXRD) and high energy radiation to measure the residual stress in a bar of uranium metal. The method used for the stress measurements is the 'sin$^2 \psi$' method.

Measurements were made in reflection at a diffraction angle 2θ of 20° and over the energy range 10 to 50 keV. The accuracy of our measurements was limited by the necessity to work in reflection and their sensitivity was limited by the high absorption of uranium. However these disadvantages were partially compensated by the large quantity of information available from ED spectra and the resolution and high intensities achievable with synchrotron radiation. The final accuracy of the measurements of d-spacing changes (strain) was approximately 1 part in 10^4.

2 - Energy Dispersive X-Ray Diffraction

In energy dispersive x-ray diffraction the energy distribution of the photons diffracted at a fixed scattering angle 2θ by a sample placed in a collimated polychromatic beam of X-rays is analysed by an energy sensitive semiconductor detector. The basic equation of EDXRD is[1]

$$Ed\sin\theta = constant, \qquad (1)$$

where E is the energy of the X-rays, d the separation of the atomic planes within the sample and θ the Bragg angle. If E is expressed in keV and d in $\mathring{A}$, the $constant$ has the value of 6.19926 (keV $\cdot \mathring{A}$).

With a white (polychromatic) beam of X-rays incident on a powder sample, expression (1) shows that for a fixed value of 2θ discreet values of d will produce reflections in an energy spectrum collected by an energy sensitive detector. This is the principle of EDXRD.

When there are no sample effects causing broadening, the profile of a reflection is the convolution of the profiles of the detector response and that due to the geometry of the collimation system. If both profiles are gaussian the full width at half maximum of a reflection, ΔE_T, is given by[2]

$$\Delta E_T = \left[\Delta E_D^2 + \Delta E_G^2 \right]^{1/2} . \qquad (2)$$

ΔE_D is the intrinsic resolution of the detector system. For the the detector used in this work it was approximately 220 eV at 15 keV and 340 eV at 50 keV. ΔE_G is the geometrical

contribution and it is given by

$$\Delta E_G = (\cot \theta \Delta \theta)^2, \qquad (3)$$

where θ is half the angle of diffraction and $\Delta \theta$ the divergence of the collimation system.

For a given fractional change in the lattice parameter of the material induced by the presence of a residual strain, the relative change in lattice parameter $\Delta d/d$ is related to the change in energy $\Delta E/E$ through the expression[3]

$$\frac{\Delta d}{d} = -\frac{\Delta E}{E}, \qquad (4)$$

obtained by differentiating (1) whilst keeping θ constant. This shows that in EDXRD a given value of $\Delta d/d$ causes the largest shifts in the positions of the reflections at high energies. The condition expressed by (4) is obviously also valid for adjacent reflections corresponding to d-spacings separated by Δd. Their separation ΔE will be largest at high energies. This increase in the sensitivity of measurements of lattice paramenter changes at high values of E is an important feature of this technique.

3 - Stress Measurements with EDXRD

In the work reported here we have only considered the elastic deformation of the crystallites caused by a *uniform* strain. Plastic deformations, which cause nonuniform microstrains, have been ignored. When present in a material they cause a broadening of the diffraction lines, an effect which cannot be measured with sufficient accuracy in EDXRD except when using standard calibration samples with a high crystalline symmetry[3].

If a residual stress is present in a polycrystalline material, the d-spacings of the crystallites are changed from their stress-free value to a new value which is related to the magnitude of the stress[4,5]. In EDXRD these changes will cause shifts of the positions of the reflections, on the energy scale, from which the strain can be calculated and the residual stress determined using the elastic constants of the material[6,7]. Hence the *stress* is not measured directly, it is the *strain* that is measured; the *stress* is determined indirectly by calculation.

The technique used here to measure residual stress is the 'sin$^2 \psi$' technique[5,6,9,10,11] where the residual stress is calculated from the measurements of the strains which are developed in directions inclined to the principal stress. When the 'sin$^2 \psi$' technique is combined with EDXRD, these strains are evaluated from the small changes in the energies of the reflections when the families of planes corresponding to these reflections are inclined

at different angles to the principal stress axis. These inclinations are achieved by tilting the sample in the plane of diffraction with respect to the normal to the diffracting planes. The tilt angle ψ can be positive or negative, corresponding to increasing or decreasing the angle between the incident beam and the surface of the sample, respectively. A diagram illustrating the geometry of the technique is shown in Figure 1. In the simplest case considered here, the measured stress is parallel to the surface of the material and the values of the d-spacings calculated when the tilt angle ψ is equal to $0°$ are used as references (diffracting planes perpendicular to the surface normal). The strains measured at different inclinations from the surface of the material are used to calculate the surface stress state using elasticity theory[11]. The surface stress state is then determined from the strains measured at different inclinations to the surface of the material using elasticity theory[11].

The relationship between strain (change in d-spacing), stress and tilt angle ψ is

$$Strain_\psi = Material.constant \times Stress_\phi \times \sin^2 \psi$$

or explicitly[11]

$$\frac{\Delta d_\psi}{d_{\psi=0}} = \frac{1+\nu}{E} \times \sigma_\phi \times \sin^2 \psi, \tag{5}$$

where Δd_ψ is the difference between the d-spacing of a family of planes measured at the tilt angle ψ and that measured at $\psi = 0°$ $(d_{\psi=0})$. ν and E are the Poisson ratio and Young modulus of the sample, respectively, and σ_ϕ is the stress parallel to the surface of the sample in a direction ϕ with respect to the principal axis of the stress state that exists in the sample. Expression (5) shows that a linear relationship exists between $\Delta d_\psi / d_{\psi=0}$ and $\sin^2 \psi$, hence the stress σ_ϕ can be calculated from the gradient of the straight line obtained by plotting the strain measured for a range of values of the tilt ψ against $\sin^2 \psi$.

In the simple approach used here, we measured the residual *single* stress in the direction of the longest dimension of the uranium bar (the *principal* axis, hence $\phi = 0°$). Generally stress is in the form of a three dimensional tensor, but such a full analysis was outside the scope of this work. (For more advanced forms of stress analysis, see [9] and references therein).

The two main advantages of using EDXRD with the '$\sin^2 \psi$' method are the fixed geometry and the wide range of energies available. As the angle of diffraction is fixed throughout the measurements, there is no scanning and the geometry is the same for all the reflections; this eliminates the need for geometrical corrections. Data are collected from many families of planes at the same time and hence the quantity of information collected is much greater than in the conventional one wavelength scanning technique; further, as all the reflections are

spread over a wide range of energies, the technique can be depth sensitive. Unfortunately, this last feature could not be exploited here as the absorption of uranium was too high.

4 - Measurements and Results

The measurements reported here were carried out on station 9.7 of the SRS using the 5 Tesla wiggler which produces a very intense beam of highly collimated high energy radiation. Because of the high absorption of uranium our measurements were made in reflection. The diffraction angle 2θ was chosen as $20°$, a compromise between optimum resolution and accuracy of stress measurements. In EDXRD, optimun resolution is obtained at low angles of diffraction using high energy radiation, but such angles limit the large range of tilts required to achieve a high accuracy with the '$\sin^2\psi$' method.

The cross section of the incident beam was defined by a 0.1 mm pinhole placed 75 cm before the sample. The divergence of the incident beam was approximately 0.1 mrad in the plane of diffraction. The geometrical contribution to the resolution was determined mostly by the divergence of the diffracted beam collimation system, two 50 cm long molybdenum bars[12]. The total divergence ($\Delta\theta$ in expression (3)) was 1.1 mrad, giving geometrical contributions (ΔE_G) of 94 eV and 312 eV at 15 keV and 50 keV, respectively. Once combined with the detector contributions given in section 2 (using expression (2)), the widths of the reflections were 240 eV and 460 eV at these energies, giving a final resolution $\Delta E/E$ of 1.6% at 15 keV and 0.9% at 50 keV.

The diffractometer was calibrated using a flat sample of Cr_2O_3, the spectrum obtained is shown in **Figure 2**. The fixed angle of diffraction 2θ was determined as $19.8652° \pm 0.0066°$. This value is the weighted mean of all the values of θ calculated using equation (1) from the energies of 32 reflections. The latter were determined by fitting gaussian envelopes to all the reflections using a peak search/fit program[13].

The sample of uraniun consisted of a 5 cm long section of the bar supplied by the Army Materials and Mechanics Research Center. The residual stress measurements were made in reflection from the narrowest flat surface with the axis of the bar parallel to the plane of diffraction. The sample was mounted on a small goniometer, aligned parallel to the incident beam and then rotated through $9.93°$, half the angle of diffraction. A spectrum was collected at this symmetric reflection position. This procedure was then repeated and the energies of the reflections were taken as the mean values of the positions calculated from the two spectra. These data, obtained with the diffracting planes parallel to the surface

of the bar ($\psi = 0°$), provided the reference values of the d-spacings used to calculate the strain as a function of tilt ψ. Spectra were collected at 10 different values of ψ within the range of $\pm 7°$. The spectra obtained at $\psi = +5.5°$ and $\psi = -5.5°$ are shown in **Figure 3**. The differences in the intensities of the reflections between the two spectra were probably due to the inhomogeneity of the sample surface caused by texture, variations in the thickness of the oxide layer and orientation effects[14]. As the ψ axis did not coincide with the θ axis, the sample position had to be reoptimised after every tilt change and hence the new spectrum was collected from a slightly different part of the sample.

Uranium has an orthorhombic structure with space group Cmcm. The theoretical positions of all the reflections were computed using the cell parameters $a_o = 2.854\text{Å}$, $b_o = 5.869\text{Å}$, $c_o = 4.955\text{Å}$ and are shown in Figure 3. The experimental reflections which do not match these positions were either fluorescent lines from uranium or reflections from U_3O_8 and UO. Some of the reflections have been labelled in Figure 3 to illustrate the complexity of the spectra. At energies above 25 keV, the number and proximity of the reflections were too high for the resolution of the technique used here and it was impossible to deconvolute the individual reflections from uranium or separate them from those due to U_3O_8. This was ignored in the data analysis and the resulting 'composite' reflections were treated as single peaks of a U - U_3O_8 mixture. Although incorrect, this procedure was found to be satisfactory. This is illustrated in **Figure 4**. We have plotted the individual values of $\Delta d_\psi/d_{\psi=0}$ calculated from the energies of all the reflections which could be fitted with gaussian envelopes in the spectrum collected at $\psi = -5.5°$ (Figure 3 (a)). The consistency of the data is very good over the whole energy range and the calculated mean value of $\Delta d_\psi/d_{\psi=0}$, $-0.76 \times 10^{-4} \pm 0.44 \times 10^{-4}$, shows that even in non-ideal experimental conditions, strains of less than 1 part in 10^4 can be detected and accurately measured. The limitations of the technique which had to be used here were partly compensated by the wide range of energies, hence the large number of reflections, which could be used to calculate the strain at every value of ψ.

The data used to determine the residual stress are shown in **Figure 5** where the values of $\Delta d_\psi/d_{\psi=0}$, calculated from the energies of the reflections using expression (4), are plotted against $\sin^2 \psi$. The straight line drawn is the result of a weighted least squares fit to the data. From its gradient and the elastic constants of uranium[15], the residual stress was calculated using expression (5) as

$$\sigma_\phi = -1097 \pm 160 \text{ MPa} \quad .$$

This value indicates the presence of a large *compressive* residual stress in the surface of the bar which is greater than the tensile strength of uranium metal (580 MPa[16]). Such high surface stresses are often found in coatings[11]. In our sample this was probably the oxide layer.

5 - Conclusion and Discussion

Using synchrotron radiation, we have shown that EDXRD and the 'sin$^2\psi$' method of residual stress measurement can be successfully combined. Lattice strains of less than 1 part in 10^4 were measured with an accuracy of 50% or better. Serious limitations were imposed on our measurements by the high absorption of uranium. **Table 1** shows the 1/e penetration depth $t_{1/e}$ for x-rays in uranium as a function of energy. It corresponds to $\mu t = 1$ in the expression for the attenuation of a beam of radiation in matter ($I = I_o \cdot e^{-\mu t}$) and represents the thickness of material which reduces the intensity of the incident beam to 1/e of its original value. These data show that measurements could not be made in the bulk of the material with the range of x-ray energies available. Hence they had to be made in reflection from a thin layer of the surface containing oxide. Further, as uranium has an orthorhombic structure, ideal experimental parameters are high energy synchrotron radiation and low angles of diffraction in order to achieve optimum resolution[2]. In reflection geometry these conditions limit the range of tilt ψ, hence lower the accuracy of the calculation of the residual stress. This also reduces the depth of penetration further. **Table 2** shows the effective penetration depth T_ψ as a function of tilt ψ T_ψ for the angle of diffraction used here. It is given by the expression[17]

$$T_\psi = \frac{\sin^2\theta - \sin^2\psi}{2\mu\sin\theta\cos\psi}$$

where μ is the linear absorption coefficient at a given energy. It can be seen that *in the work reported here the maximum value of T_ψ was approximately 5 μm at 50 keV.*

Faced with these limitations, the simplest form of analysis was chosen: the 'sin$^2\psi$' method with the assumptions of a single-axis stress state[11] (one value of ϕ) and of no stress component in the direction of the surface normal[17] ($d_{\psi=0}$ as reference). We also ignored the probable anisotropy of the elastic properties within the individual crystallites resulting from the interaction between the measured crystal strains and the surface stress. Hence we used the elastic constants predicted by isotropic elasticity theory from the bulk modulus[15,18].

6 - Future Work

A high voltage generator (150 to 300 kV) would be needed to perform stress measurements on uranium if a reasonable penetration is required. Without this, all measurements are limited to a very thin surface layer (see Tables 1 and 2). Ideally, a 300 kV generator should be used for bulk stress measurements on thin samples ($\leq$ 0.5 mm) using transmission geometry. Alternatively, measurements can be made using neutrons[15], but without the possibility of making the experimental setup portable.

All restrictions become less severe as the atomic number of the materials are reduced, to tungsten through to iron, for example. This is illustrated in Table 1 where the 1/e penetration depth of x-rays in iron is also given. Stress measurements, mapping are currently being made on iron using synchrotron radiation from the SRS wiggler, transmission geometry and samples up to 15 mm thick. Such measurements could also be made using a high voltage generator, but with samples only a few millimeters thick to compensate for the large reduction in the intensity of the radiation. Detailed stress analyses which have only been performed on sample surfaces using the angle scanning mode [14,19,20,21,22] could then be made inside materials using transmission geometry at a fixed angle of diffraction. This last feature would greatly facilitate the design of a portable system (portable systems using angle scanning are described in references [10] and [18]).

Daniel Häusermann, 30 June 1988

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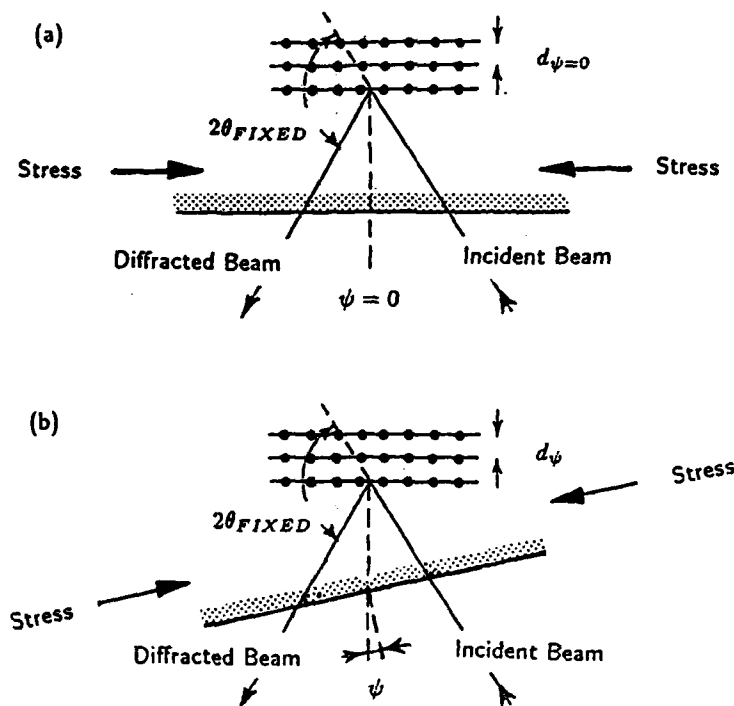[23] J.H. Hubbell, Int. J. Appl. Radiat. Isot., 1982, Vol. 33, 1269-1290.

Figure 1 - Diagrams illustrating the geometry of the $\sin^2 \psi$ technique in the energy dispersive case. If a compressive residual stress exists in a material, the separation between the planes normal to the stress axis is decreased whereas it is increased for those parallel to that axis. Consequently strains in the crystal structure of the material are not only confined to the direction of the residual stress and the d-spacings of identical crystallographic planes oriented at different angles ψ to the surface will be changed by different amounts. In (a) the sample tilt ψ is zero and the diffracting planes are parallel to the sample surface. The energies of the reflections in a spectrum collected at a fixed angle 2θ are used to calculate reference values for the d-spacings of the various families of planes in the material. In (b) the sample is tilted by an angle ψ with respect to the normal to the diffracting planes. A spectrum is collected at the same fixed angle 2θ, but the energies of the reflections are changed as the d-spacings are now affected by the component of the stress parallel, as well as perpendicular, to the sample surface. From these changes in energy, the residual stress can be calculated.
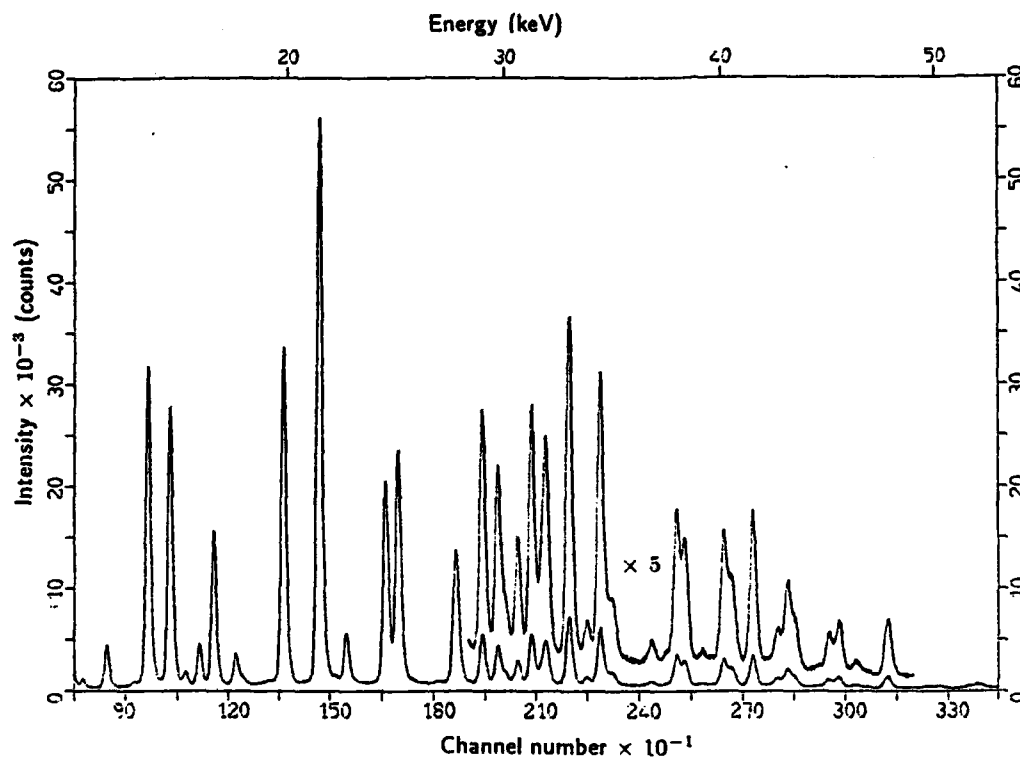
Figure 2 - Energy dispersive spectrum of Cr$_2$O$_3$ used to calibrate the diffractometer. This spectrum was collected in reflection from a rotating powder sample in 45 minutes with the SRS operating at 2 GeV and a mean current of 115 mA. The angle of diffraction 2θ was calculated as 19.8652°± 0.0066° using 32 reflections.

Energy (keV)

Intensity × 10⁻³ (counts) — $\text{Intensity} \times 10^{-3}$ (counts)

(a)

$U_{(110,021.002)}$

$U_{(020)}/U_3O_8$ $U_{L\alpha2}/U_{L\alpha1}$

$U_{L\beta1}/U_3O_8$

$U_{(022)}$

$U_{L\gamma3}/UO$

× 10

U/U_3O_8

Calculated positions of the reflections for Uranium

(b)

$U_{L\gamma1}/U_{(112)}$

$U_{(131)}/U_3O_8$

$U_{L\beta2}/U_3O_8$

U/U_3O_8

$U_{(111)}$
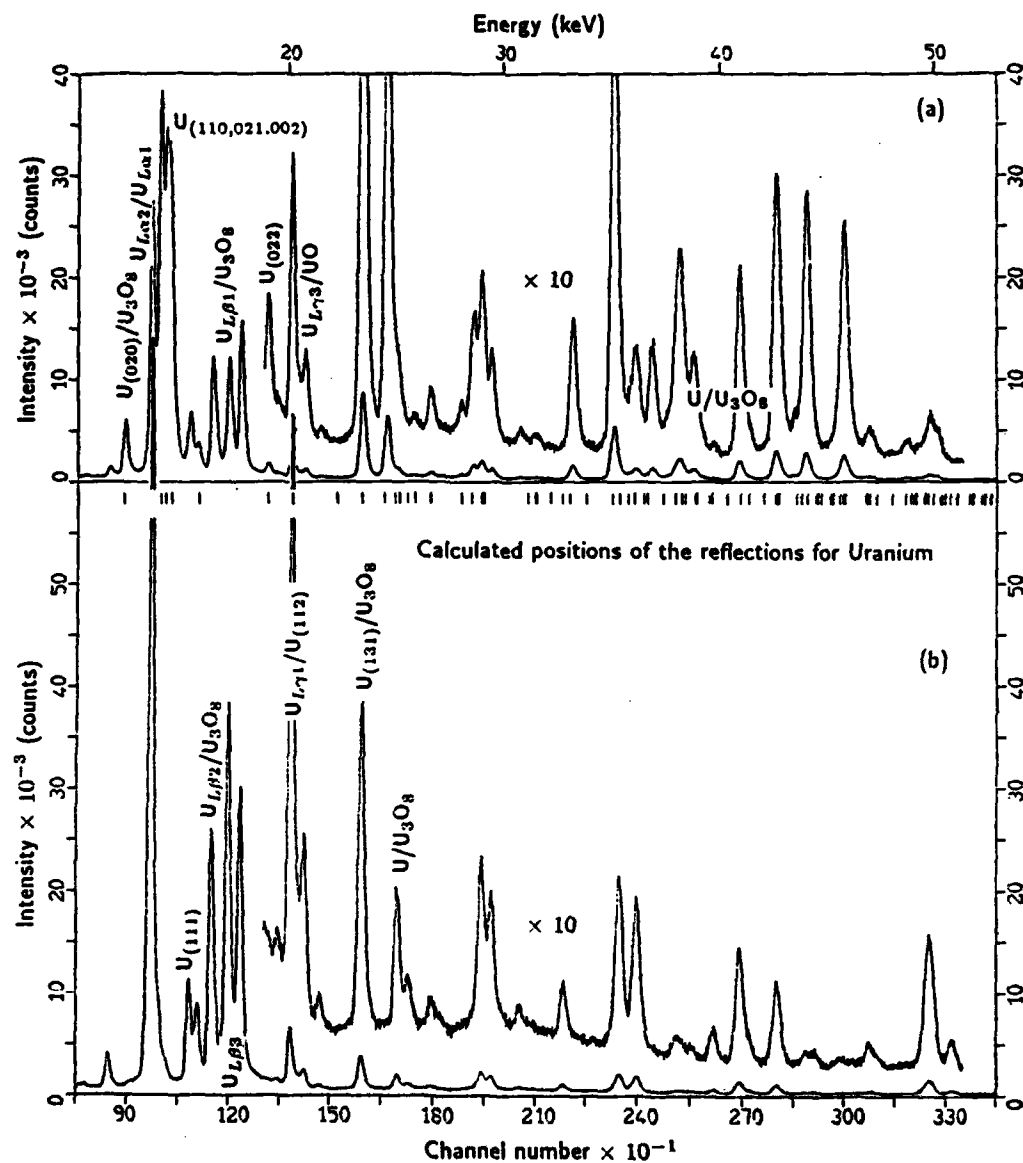
$U_{L\beta3}$

× 10

Channel number × 10⁻¹

Figure 3 - Two spectra used to calculate the residual stress. The tilt angle ψ were $-5.5°$ and $+5.5°$ for (a) and (b) respectively. Both spectra were collected in 30 minutes at 2 GeV and a beam current of approximately 100 mA. The calculated positions of the reflections for uranium at $2\theta = 19.8652°$ are shown for comparison.
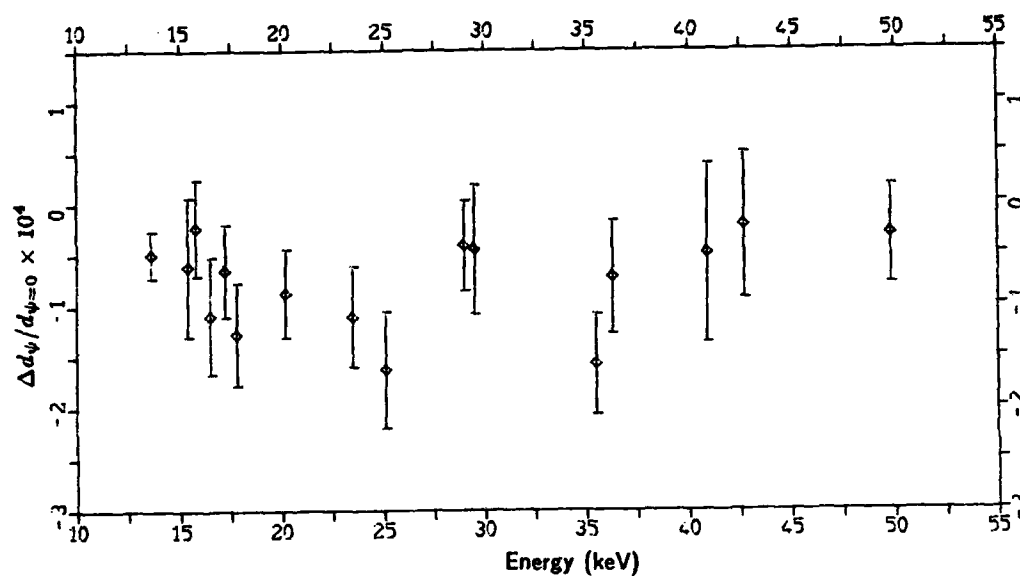
Figure 4 - Values of $\Delta d_\psi / d_{\psi=0}$ obtained from all the reflections in the spectrum collected at $\psi = -5.5°$. The mean value of $\Delta d_\psi / d_{\psi=0}$ is -0.76×10^{-4} with a standard deviation of 0.44×10^{-4}.
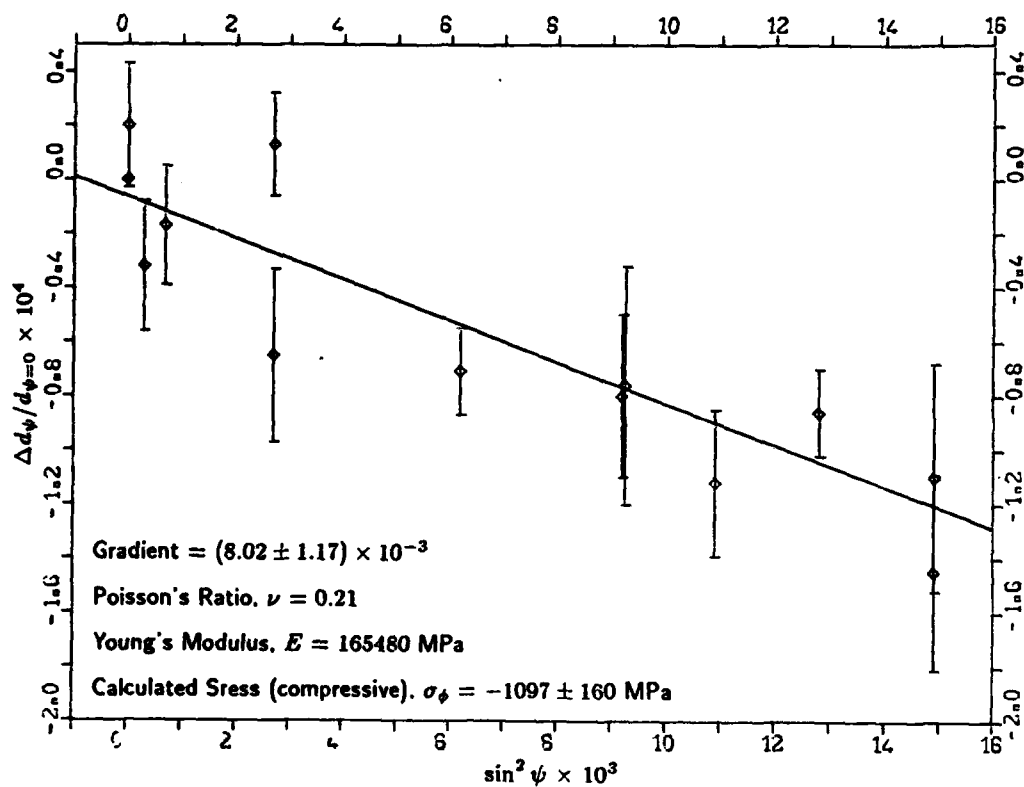
Figure 5 - Strain versus $\sin^2 \psi$. The values of $\Delta d_\psi / d_{\psi=0}$ plotted are the means calculated from up to 15 reflections in a spectrum. The tilt ψ was varied from -7.0° to $+7.0^\circ$. The straight line is the result of a least-squares fit. The values of the elastic constants for uranium were taken from reference [15].

Table 1 - The $1/e$ penetration depth $t_{1/e}^{(1)}$ for x-rays in uranium and iron as a function of energy. The absorption data were taken from reference [23].

E (keV)	$t_{1/e}$. U	$t_{1/e}$. Fe
8[2]	1.7 μm	4.2 μm
15	8.0 μm	22 μm
20	7.4 μm	50 μm
40	26 μm	350 μm
60	75 μm	1.1 mm
80	160 μm	2.2 mm
100	270 μm	3.4 mm
150	200 μm	6.5 mm
200	400 μm	8.7 mm
300	1.0 mm	12 mm

(1) see text for definition

(2) $Cu_{K\alpha}$

Table 2 - The effective penetration depth $T_\psi^{(1)}$ in μm for uranium as a function of sample tilt ψ for different x-ray energies at $2\theta = 20°$. The parameter μ_{abs} is the absorption coefficient. the values were taken from reference [23].

Energy (keV)	10	15	30	45	60
μ_{abs} (cm^{-1})	3410	1240	786	296	134
$\psi = 0°$	.25	.69	1.1	2.9	6.4
$\psi = \pm3°$	.23	.63	1.0	2.7	5.9
$\psi = \pm5°$	.19	.52	.82	2.2	4.8
$\psi = \pm7°$	.13	.35	.55	1.5	3.2

(1) see text for definition